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Marble Durability Assessment by Means of Total Optical Porosity and Adjacent Grain Analysis / Bellopede, Rossana; Zichella, Lorena; Marini, Paola. - ELETTRONICO. - 848:(2020), pp. 35-47. [10.4028/www.scientific.net/KEM.848.35]

Availability:

This version is available at: 11583/2836047 since: 2020-07-17T11:40:01Z

Publisher:

Trans Tech Publications

Published

DOI:10.4028/www.scientific.net/KEM.848.35

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Marble durability assessment by means of total optical porosity and adjacent grain analysis.

Rossana Bellopede^{1, a *}, Lorena Zichella^{1, b}, Paola Marini^{1, c}

¹Politecnico di Torino, DIATI, Corso Duca degli Abruzzi, 24, 10129, Torino, Italy.

^arossana.bellopede@polito.it, ^blorena.zichella@polito.it, ^cpaola.marini@polito.it

Keywords: total optical porosity, adjacent grain analysis, decay, fabric, porosity.

Abstract. The presence of pores, cracks and microcracks in marble is one of the main features that govern the processes of decay of this stone material and, although marble is characterised by a modest porosity, there is a clear correlation between the presence and movement of fluids, and the phenomena of alteration. Through the study of porosity, it is possible to better understand the phenomena of alteration and degradation in order to obtain useful information, not only in the field of modern building, but also for the protection and recovery of historical and artistic heritage goods. This study was conducted through the characterisation of parameters directly related with the degree of alteration of the materials: water absorption at atmospheric pressure (EN 13755), open porosity (EN 1936), flexural strength (EN 12372) and bowing (EN 16306 par. 8.2). The physical and mechanical measurements have been compared with the Total Optical Porosity method (TOP) and the Adjacent Grain Analysis (AGA) index (a suggested method to evaluate the marbles' tendency to bow, in EN 16306 annex C); two different methodologies both based on image analysis. The purpose of this study is to demonstrate the effectiveness, for the assessment of marble durability, of the two techniques of microscopic image analysis, the first correlating to the grain shape and the second to the open porosity index. This was done by comparing the microscopic image analysis results with the physical and mechanical properties, both after artificial ageing and after ten years of natural ageing. The results obtained with the TOP method seem to represent the tendency to decay better than the AGA index. The comparison of image analysis of the thin sections, in different portions of the marble specimens, shows the development of degradation due to atmospheric agents, from the surface to the inside, of naturally aged specimens, confirming recent studies made on different marbles.

Introduction

Marble is suitable for a wide range of uses in the construction sector. It can be used as an architectural element for urban and interior decoration, as well as for interior and exterior cladding, as it shows excellent mechanical and aesthetic characteristics. The main problem, related to the use of this ornamental stone, is the decay effect. The factors causing the degradation of marble can be both intrinsic and extrinsic. The first are related to the physical-mineralogical characteristics of the stone, i.e. its porosity, mineralogy and structure. The latter are linked to external factors, such as the temperature, pressure and relative humidity, to which the stone is subjected. These degradation factors produce a worsening of the mechanical properties of the material [1], such as an increase in water absorption and a decrease in flexural strength, due to the increased porosity of the rock, and in some cases, mainly in marble, can be linked to bowing. According to several studies carried out, the shape of grains appears to be one of the factors that most influence the deterioration of marble and can be indicative of its durability [2]. The relationship between structure and mechanical properties was studied as early as 2001 and then in 2003 [3,4] using the image analysis technique, by measuring the perimeter of the mineral phases and determining the specific surface area using optical microscopy to quantify the grain dimension. The first results obtained seem to confirm that the study of the texture, by means of the image analysis, could be a valid tool for determining the

durability of the rocks, even if there was a need to define the test methodology and to define the relevancy of the methods.

A good correlation between the number of adjacent grains (AGA), and therefore the grain shape (granoblastic and xenoblastic), and the durability, was confirmed in a 2006 study [5]. Marble with polygonal grains (low AGA index) has tendency to bow, in contrast to marble with allotriomorphic grains (high AGA index – no tendency to bow) [5]. In other recent studies, further claims in relation to AGA and marble durability have been made. In a study focussed only on natural ageing and a marble with grain size lower than 0.5 mm, a direct relation between flexural strength, after natural ageing, and AGA values has been found [6]. Widening the research field, onto artificially aged stones, even with a mean grain size > 0.5 mm, the AGA index was not always in correlation with the physical mechanical properties of the marble (bowing, flexural strength and UPV) [7].

Concerning the physical/mechanical evaluation of marble decay, recent works regarding microscopic characterisation, carried out by means of the Image Analysis method [8,9], have found a direct connection between durability and structural characteristics, observable by microscopic analysis (microcrack development and calcite anisotropy). In recent works a good correlation between open porosity (detected by means of microscopic image analysis) and standardised water absorption measurement of the marble, was detected using artificially aged marble [7,10].

Another method to measure porosity is the contact sponge [11] but results of this methodology are very dispersed and for this reason it was not used [7,10]. However natural decay, on 10-year exposed slabs, did not match with the artificially aged ones [6] as flexural strength, water absorption and bowing showed different trends.

The purpose of this study is to assess the reliability of the AGA index in the detection of marble durability and to optimise the methodology to evaluate the use of TOP in the study of marble decay, by comparing natural and artificial ageing. In fact, the study of the development of porosity from the external to the internal side of a slab, analysed with TOP, could explain the differences in behaviour of a marble subjected to a slow (natural) or to a rapid, artificial, weathering.

Materials and Methods

The five marble samples tested, and their petrographic characteristics, are shown in Table 1. All the tested marbles are totally calcitic. Three of them are characterised by a polygonal grain shape: GI, PS, SG, PS and SG, at difference of GI have a higher grain size and a seriate structure. According to previous literature [3,4,5,12], all these three marble samples could be potentially unsafe due to the grain shape, but this should be confirmed by bowing, after the thermal cycles in the presence of humidity.

Marble Type	Origin	Structure	Presence of subgrain	Grain size [mm]	Tendency to bow according to [3,4,5,12] assertions
GI	Italy	polygonal and equigranular grains	no	0.1-0.5	yes
PS	Portugal	seriate polygonal	yes	1-2.8	yes
CA	Italy	interlobate/equigranular	yes	0.01-0.3	no
RE	Portugal	allotriomorphic weakly interlobated irregularly	yes	0.1-0.9	no
SG	Portugal	seriate polygonal	no	0.7- 1	yes

Table 1: Tested marble with their petrographic description.

Figure 1 shows the microscopic image of the thin sections of the studied marble.

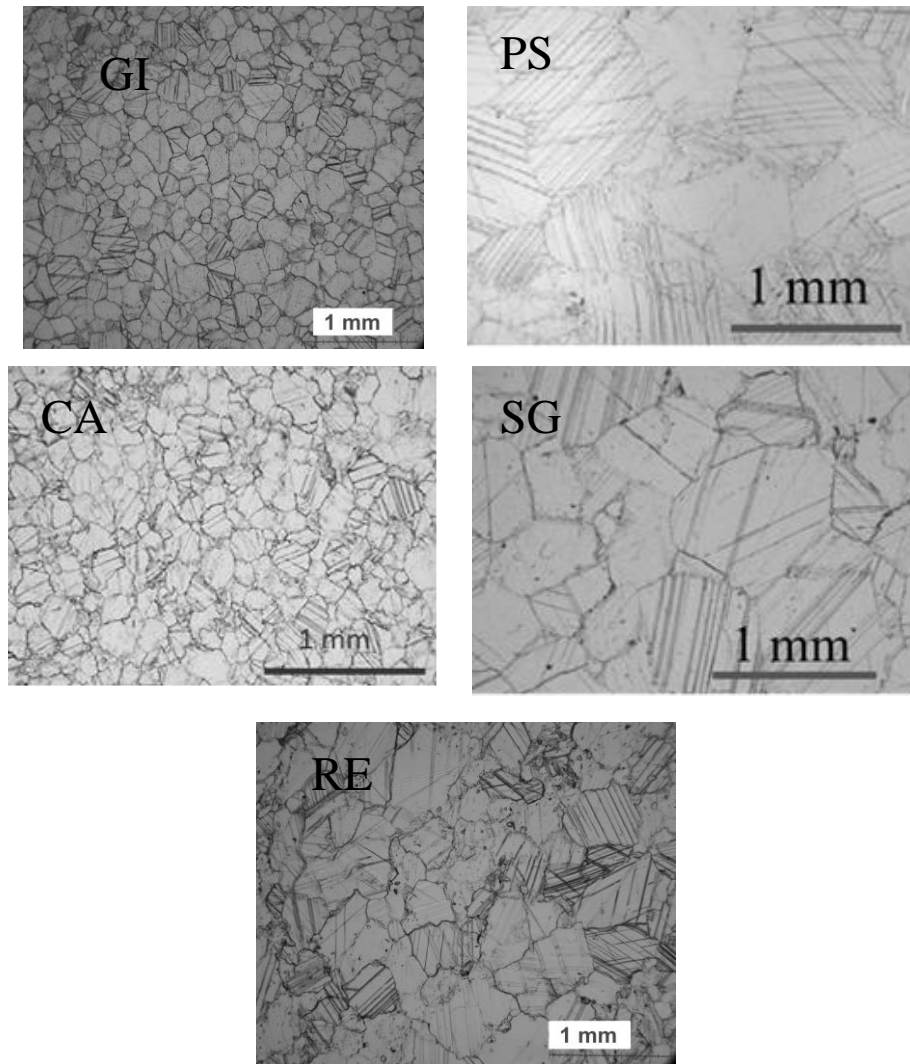


Figure 1: Microscopic photo of all the tested marbles. From left top: GI marble, PS marble, CA marble, SG marble and RE marble. Photos author(s)

The specimens have been tested in natural conditions, after the artificial ageing test (after 50 thermal cycles in presence of humidity according to EN16306), and after natural ageing for 10 years. Table 2 shows all the tests conducted, for the three measurement conditions, for all the considered marbles.

Test condition			Natural condition					Natural ageing					Artificial ageing				
Marble tested			CA	GI	PS	RE	SG	CA	GI	PS	RE	SG	CA	GI	PS	RE	SG
Test performed	Image analysis	AGA	X	X	X	X	X	-	-	-	-	-	-	-	-	-	-
		TOP	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X
	Physical and mechanical tests	UPV	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X
		Open porosity	X	X	X	X	X	-	-	-	X	-	X	X	X	X	X
		Bowing	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X
		Flexural strength	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X
		Water absorption	X	X	X	X	X	X	X	-	X	-	X	X	X	X	X

Table 2: List of tests performed for all marbles.

Total Optical Porosity method

Total Optical Porosity (TOP) determination was conducted using the free software IrfanView 4.40 and the macro file jPOR.txt [13] for ImageJ. Five different kinds of marble were tested. For each one, six thin sections were made and soaked with epoxy resin and methylene blue as illustrated in Fig. 2a. In order to obtain a better analysis of the increase of decay, five thin sections were cut from the weathered marble, respectively three along the upper (PAR1), medium (PAR2) and lower (PAR3) sections parallel to the exposed surface and one along a transverse section (TRASV). The impregnation process was conducted, repeatedly, under vacuum in order to obtain a smooth surface, when viewed against light. All the thin sections were analysed using the optical microscope LEICA MZ6 and photographed by means of the PANASONIC LUMIX DMC-GF6 digital camera. For each thin section, ten photos were taken, uniformly spaced along its surface (Fig. 2b), and then pre-processed using IrfanView. Each 24-bit image was converted to an 8-bit image, using the custom blue palette of JPOR created, in order to reach a colour threshold that requires less pre-processing and removal of noise, thereby reducing the inter-operator variability. The threshold values used to determine the porosity were left as constant as possible (lower threshold = 0 and upper threshold = 69/70) in order to yield representative and comparable results between the different marbles. At the end of this process, the average porosity value was calculated and compared, for each thin section, as a percentage of blue stained area with respect to the whole photo area. Fig. 3 shows an example of the image process analysis for the selection of porosity.

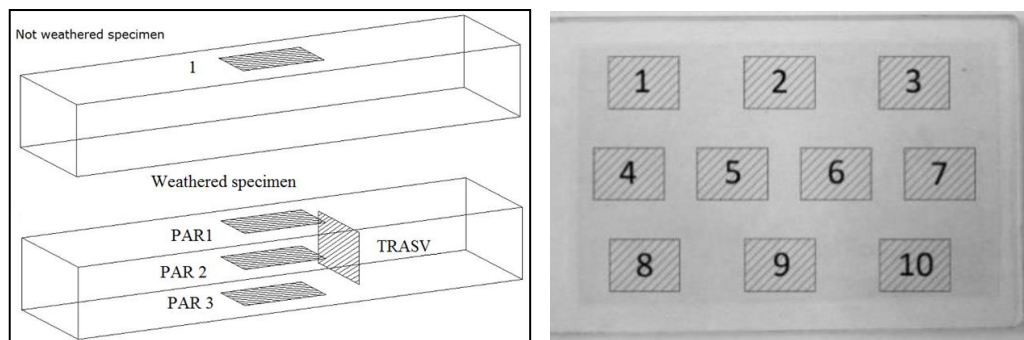


Fig. 2: a) left: areas cut from the specimens to obtain thin sections. b) right: Position of pictures taken from the thin section for image analysis.

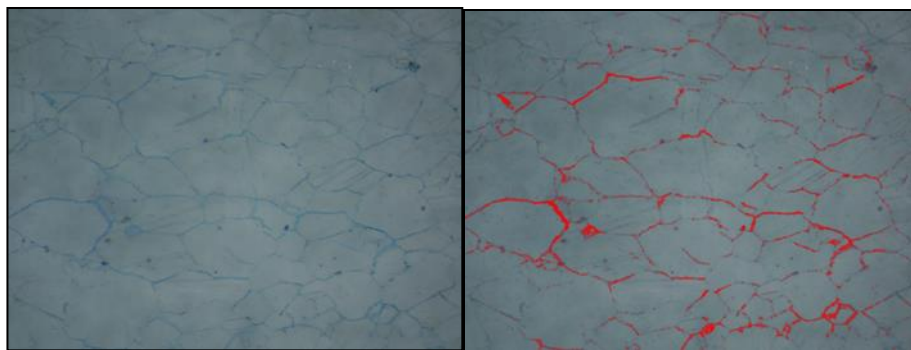


Fig. 3: Example of porosity selection using image analysis Left: original image. Right: image elaboration with selection of porosity. Photos author(s)

This new, unconventional, technique was used to verify if the marbles showed any differences in porosity in different sections of the specimen after being subjected to both natural ageing and to accelerated ageing tests [7]. For the GI marble, even if the values on the TOP are characterised by

high uncertainty, it is possible to detect differences, in terms of porosity distribution, between artificial and natural weathering, which would explain the different results obtained from the conventional tests (with particular reference to water absorption and bowing values).

AGA method

The Adjacent Grain Analysis (AGA) was carried out in accordance with the EN 16306 Annex C (2013) by means of the free software ImageJ in order to calculate the number of adjacent grains (AG) around median-sized grains. AGA methods were performed, in the natural condition only, for all stone tested. This analysis method gives information on the microstructure of natural stone and its potential durability [12] and it has been suggested as a possible screening method. Initially, the value of the median grain size was calculated by measuring the Feret diameter (longest axis) of at least 100 grains along the plotted linear traverses (Fig. 4). Afterwards, at least 50 median sized grains were chosen and a manual count of the number of their adjacent grains was carried out. The mean value of all the counts represents the AGA index. According to Annex C of EN 16306, values from eight upwards represent marbles with good characteristics, an AGA of seven is intermediate, and six indicates a low-level material. It is important to highlight that, according to the standard, this classification is valid only for calcitic marbles.

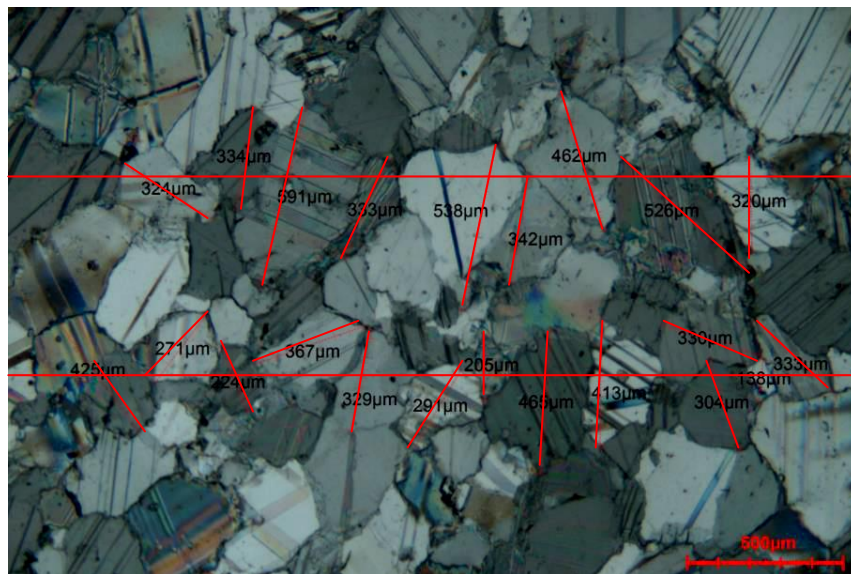


Fig. 4: Linear traverse method for determination of the grain size distribution. In red: two lines longest axis: Ferret diameter measures; In red: lines on grains: transverse diameter. Photo author(s)

Fig. 5 shows a scheme, from EN 16306 annex C, representing two types of marble, one with a grain-block structure, and one with an interlobate structure. As can be seen, the granoblastic structure has a rather low AG value, while the one with an interlobate structure has a very high AG value. This index corresponds to the tendency of marble to bow. In fact the marbles with an AGA value equal to or less than six are considered very susceptible to bending while the marbles with AGA values equal to or greater than eight have an excellent resistance to bending.

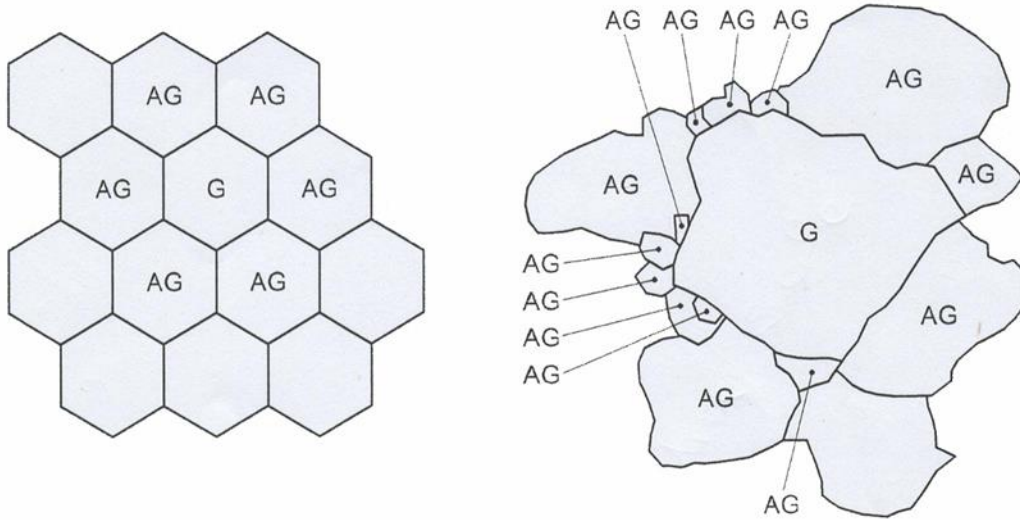


Fig. 5: Representation of the AGA calculation of different marble structures: Left: example of a granoblastic structure; Right: example of an interlobate structure. Source: EN 16306 annex C.

Physical and mechanical measurements

The physical and mechanical characterisations carried out on the five samples are listed in Table 2.

Water absorption and open porosity

The water absorption, at atmospheric pressure (WA), was conducted according to EN 13755. It is expressed as a percentage and is calculated by means of Eq. 1:

$$Ab [\%] = (m_s - m_d) / m_d * 100 \quad (1)$$

Where: m_s is the saturated mass of the sample [g], and m_d is the dry weight of the sample [g].

The open porosity, according to EN 1936 is calculated by means of Eq.2:

$$p_o [\%] = (m_s - m_d) / (m_s - m_h) * 100 \quad (2)$$

Where: m_s is the wet sample mass [g], m_d is the dry sample mass [g] and m_h is the sample mass immersed in water [g].

As the measuring is performed on specimens of dimensions 50x50x50mm by immersion in water and not by microscopic observation (as TOP), it is expected to obtain values of open porosity different from total optical porosity.

Flexural strength

Flexural strength, carried out according to EN 12372, is expressed in [MPa] and is calculated by means of Eq.3:

$$\sigma \text{ [MPa]} = (3 \cdot F \cdot L) / (2 \cdot b \cdot h^2) \quad (3)$$

Where: F is the breaking load [N], L is distance between the blades [mm], b is the specimen width measured by means of digital calibre [mm] and h is the specimen thickness measured by means of digital calibre [mm].

Bowing

In the laboratory, bowing is determined by means of an accelerated ageing test, performed according to EN 16306. Specific environmental conditions, in order to reproduce the effects of solar irradiation and humidity, are recreated. The system subjects samples to alternating cycles at a controlled temperature and humidity. The samples are placed in a tank with a controlled water level and exposed to InfraRed lamps. Reference standard requires a total of 50 cycles, but for the purpose of this research, 90 cycles were carried out. Each cycle lasts 24 h, during which, the temperature rises gradually, over 3 to 4 hours, up to a temperature of 80°C and then down to 20°C. The bowing value is determined by the difference between the height measured before the start of the test and the height measured at the nth cycle. The measurement is then normalised by dividing the bowing size by the length of the specimen according to Eq. 4.

$$B \text{ [mm/m]} = \Delta H / L \quad (4)$$

Where: B is the normalised bowing measure [mm/m], ΔH is the non-normalised measurement [μm] and L is the length of the sample [mm].

In-situ bowing measurements were executed by means of a bow-meter, consisting of an aluminum bar, with a digital calliper in the middle, placed on a mobile track that was able to measure the size of bowing in the centre of the slab.

Ultrasonic Pulse velocity

Ultrasound Pulse Velocity test was carried out by means of indirect method using transducers with a frequency of 33kHz. The measurements were made according to a spread consisting of 8 points, spaced 40 mm apart, while the distance from the edge is 60 mm (Fig. 6).

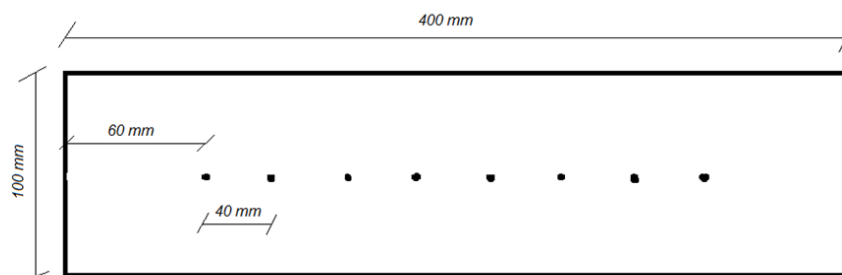


Fig. 6: Laying arrangement for each sample along which to measure the propagation velocity of the ultrasounds by the indirect method.

UPV was performed, on all the samples, before the start of the artificial aging test and after the end of the artificial cycles, in order to compare the obtained values and observe any correlation between the degradation and the propagation speed.

Results and discussion

Physical and mechanical results

All physical and mechanical characterisation tests (Table 3) were carried out before and after natural and artificial ageing, to enable comparison of the results, in order to evaluate the decay degree of each marble.

In Table 4, the results of the percentage variation in water absorption at atmospheric pressure WA, open porosity, flexural strength, UPV, and bowing after 50 thermal cycles in the presence of humidity according EN 16306 and after natural ageing, respectively, are shown. From the comparison of tables 3 and 4, there is no correspondence between the physical and mechanical properties measured after artificial and natural ageing. Artificial ageing, with 50 cycles, does not simulate a natural ageing of about 10 years. The marble sample CA showed low variations in WA, flexural strength and UPV with natural ageing as compared to artificial ageing. The ageing cycles, in this case, greatly worsen the characteristics of the marble, but the bowing values remain almost the same for both ageing tests.

	Natural condition				Natural ageing					Artificial ageing				
	WA	Open Porosity	Flexural Strength	UPV	WA	Open Porosity	Flexural Strength	UPV	Bowing	WA	Open Porosity	Flexural Strength	UPV	Bowing
	[%]	[%]	[MPa]	[m/s]	[%]	[%]	[MPa]	[m/s]	[mm/m]	[%]	[%]	[MPa]	[m/s]	[mm/m]
CA	0.03	0.16	21.5	2984	0.11	n.a.	20.60	2274	0.16	0.15	0.20	18.00	2428	0.18
GI	0.14	0.57	4.7	2282	0.52	n.a.	2.30	n.a.	0.89	0.26	0.63	2.30	1200	6
PS	0.06	0.17	16.27	2780	n.a.	n.a.	n.a.	n.a.	0.06	0.05	0.17	12.86	2559	n.a.
RE	0.13	0.34	14.2	3213	0.34	0.36	12.10	2407	0.06	0.13	0.37	14.80	2308	0.17
SG	0.07	0.17	15	2535	n.a.	n.a.	n.a.	n.a.	0.04	0.07	0.22	12.10	2062	n.a.

Table 3: Physical and mechanical properties in natural conditions (original values) , after natural ageing and artificial ageing of the marbles tested

Natural ageing - variations (%)					Artificial ageing -variations (%)			
	WA	Open Porosity	Flexural Strength	UPV	WA	Open Porosity	Flexural Strength	UPV
	105.			-				
CA	3	20.0	-16,3	18.6	49.2	n.a.	-4.2	-3.0
				-	186.			-
GI	86.7	9.5	-51.1	47.4	0	n.a.	-45.0	68.0
PS	-6.4	-4.9	-21.0	-7.9	n.a	n.a.	n.a.	n.a.
				-	174.			-
RE	5.6	10.2	4.2	28.2	2	8.0	-14.8	31.0
				-				
SG	0.0	23.0	-19.3	18.7	n.a.	n.a.	n.a.	n.a.

Table 4: Variation as percentage of the physical and mechanical properties after natural ageing and artificial ageing tests n.a. = not available

In the case of the marbles GI and RE, they show an opposite trend as compared to the marble CA. The characteristics of marbles GI and RE, after natural ageing, are worse than those after artificial ageing. Bowing values are also higher for natural ageing. These results are a confirmation of previous research [7] made on Italian marble with average grain size of 0.5mm, adding the results of a new calcitic Portuguese marble with interlobate grain habit (RE).

Image analysis

The results of the image analysis by means of AGA and TOP are reported in table 5.

specimens	AGA	TOP natural condition	Artificial Ageing				Natural Ageing			
			TOP (Par1)	TOP (Par2)	TOP (Par3)	TOP (Trasv)	TOP (Par1)	TOP (Par2)	TOP (Par3)	TOP (Trasv)
		[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
CA	9.4	0.2	1.0	1.0	0.9	1.0	0.5	0.1	1.1	0.2
GI	6.1	0.3	0.7	0.8	0.3	1.8	16.7	0.6	7.1	12.9
PS	10	0.2	0.0	0.1	0.0	0.0	n.a.	n.a.	n.a.	n.a.
RE	6	0.1	0.8	0.3	0.4	0.4	1.7	1.3	0.5	1.1
SG	6.8	0.2	0.4	0.1	0.2	0.2	n.a.	n.a.	n.a.	n.a.

Table5: AGA and Total Optical Porosity

In order to compare the different values of porosity determined for open porosity and TOP, the values of open porosity are shown in table 6.

	Open Porosity (Natural Conditions)	Open Porosity (Artificial Ageing)
	[%]	[%]
CA	0.16	0.20
GI	0.57	0.63
PS	0.17	0.17

RE	0.34	0.37
SG	0.17	0.22

Table 6: Values of open porosity in natural conditions and after artificial ageing

Through the Total Optical Porosity, it can be further confirmed that the degradation of the marbles is different regarding the natural ageing and the artificial ageing, which is illustrated by the kind of porosity variation inside the specimens. Natural degradation shows substantial differences between TOP PAR1, PAR2, PAR3 and TRASV. These differences may be related to the marble grain size. Marbles with lower average grain size show higher variation between the TOP at the external surface (PAR1) and the inner part of the specimens (PAR2). In particular the marble GI and CA show a variation respectively of 80% and 96% from the exposed side (TOP par 1) to the inner one (TOP par 2), while RE with a higher grain size (see figure 1 and table 1) has a variation of 23%. On the other side, the artificial degradation shows almost similar values of TOP for PAR1, PAR2, PAR3 and TRASV. This demonstrates the difference between the two ageing processes.

It is important to verify the correlation between the physical-mechanical characteristics of the samples analysed and the results obtained through the AGA.

Figure 6 shows the correlation between WA (Water Absorption at atmospheric pressure) and AGA value. In general, WA increased with a decrease of AGA, probably due to the increased availability of absorption surface with the decrease of sub-grains. The correlation between AGA and WA after the two different kind of decay could be due to the habit of the grain. In other words for a polygonal structure, with small (linear) contact among grains, the decay is linked to an increase of voids and therefore in water absorption. Lower is the AGA and higher is the tendency to increase in porosity and then in water absorption of sample tested mainly in the artificial ageing (with a best correlation).

In addition, considering the correlation AGA-WA, there is a high slope (degree of variation) of water absorption in natural ageing with respect to the artificial one, especially for the marbles with medium to coarse grain (GI and PS). CA, which is fine grained, shows a small difference in water absorption from the artificial ageing compared to the natural ageing.

Figure 7 shows the correlation between Total Optical Porosity (TRASV value) with water absorption. The transverse section of the specimens, soaked for the JPore analysis (see scheme in Fig. 2), is the one that best represents the whole specimen and has a better match with the water absorption. RE and GI specimens show an increase of water absorption and TOP in natural ageing compared with the artificial ageing. CA specimens instead show a little variation. It seems that grain dimensions play an important role in the artificial decay.

Figure 8 shows the good correlation with TOP and UPV value. In this case, in natural ageing, it is the PAR1 TOP value that better correlates UPV specimen value. For artificial ageing, it is the TRASV TOP value that better correlates with the UPV specimen value. This aspect is linked with the decay type. In natural conditions it is the surface of the specimens which is more affected by decay, whereas in artificial conditions, it is the whole of the specimen which is affected by decay.

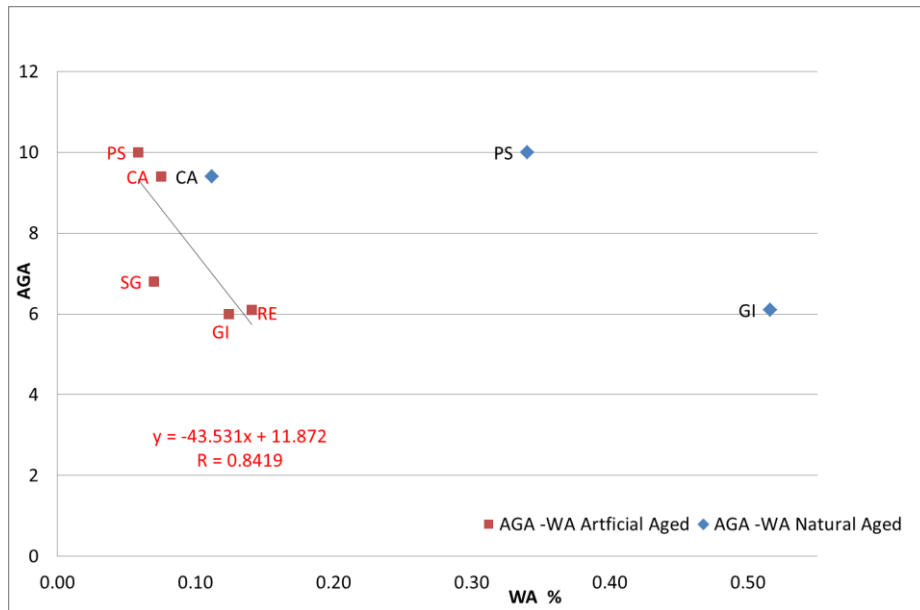


Figure 6: Correlation between water absorption at atmospheric pressure and AGA value. In red: samples and trend for artificially aged specimens. In blue: values for naturally aged specimens.

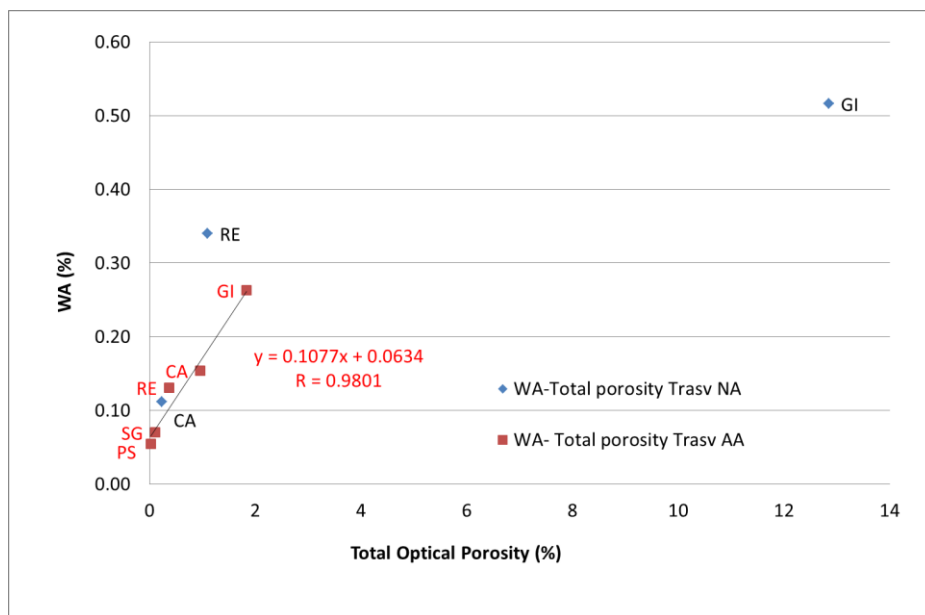


Figure 7: Correlation between Total Optical Porosity and water absorption at atmospheric pressure. In red: samples and trend for artificial ageing specimens. In blue: values for natural aged specimens.

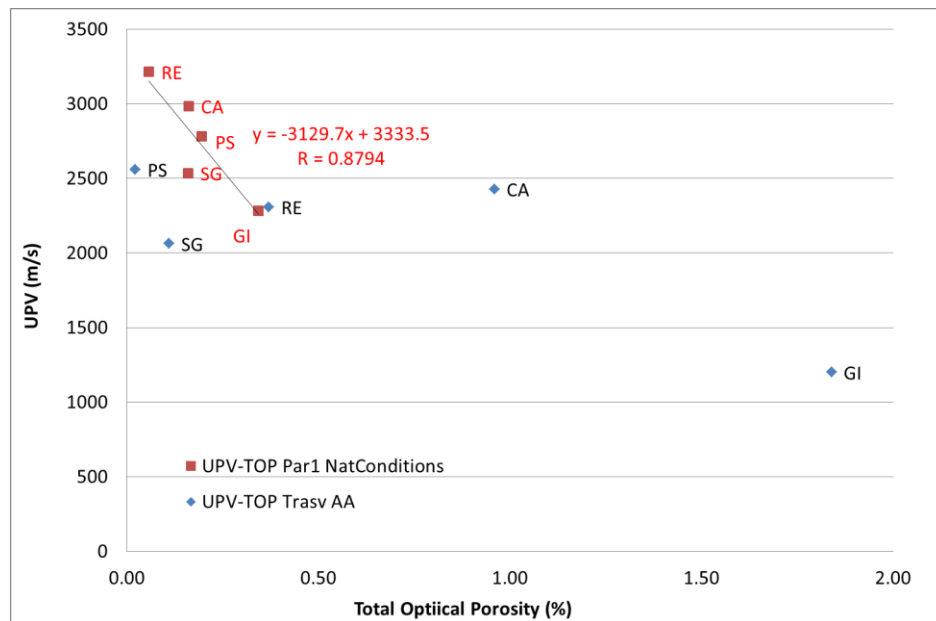


Figure 8: Correlation between Total Optical Porosity and UPV value. In red: samples and trend for artificial ageing specimens. In blue: values for natural ageing specimens.

Flexural strength correlates to the whole characteristic of the specimens and not only with the surface. Figure 9 shows the good correlation between the flexural strength and TOP TRASV value. GI is the marble sample which is more affected by natural degradation than others. This aspect is also reflected by the bowing values obtained. AGA value does not discriminate this aspect.

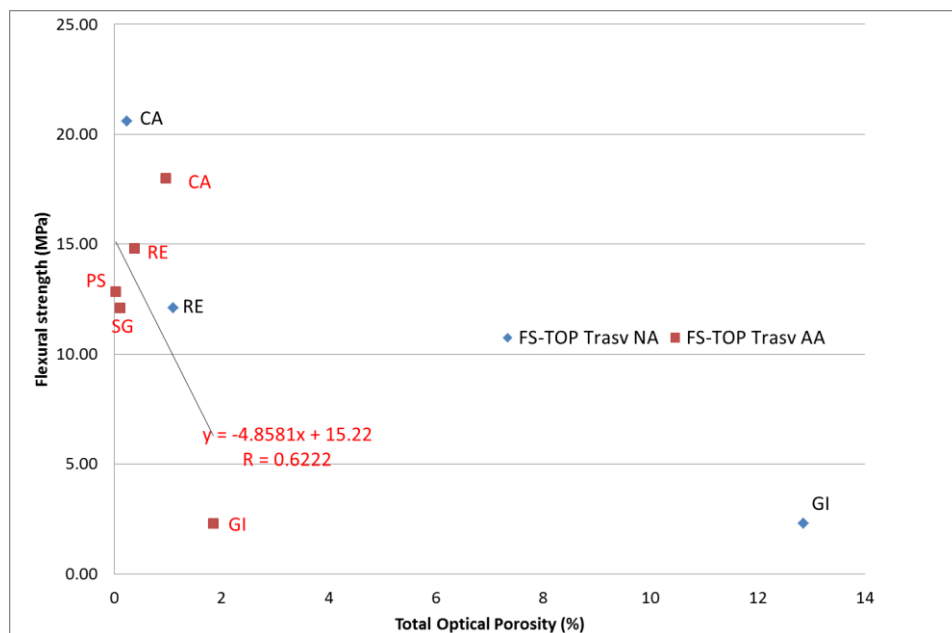


Figure 9: Correlation between Total Optical Porosity and flexural strength value. In red: samples and trend for artificial ageing specimens. In blue values for natural ageing specimens.

Discussion and Conclusions

The measure of Total Optical Porosity, on a colour impregnated thin section could be used to better explain the decay mechanism of the tested marble, in particular regarding the comparison of the

results obtained from the specimen portion PAR1 with the sections taken from the central part of specimen PAR2 as this is an indicator of the evolution of porosity or cracking inside the stone. For example, GI, with a high value of water absorption after artificial ageing (table 2), is characterised by higher values of TOP both in the upper and in central parts of specimen (PAR1 and PAR2 thin section) with respect to the TOP calculated in the natural condition ("1" thin section – table 6). The degree of variation between the external and the inner portion of specimens could be linked to the average grain size of the marble.

Furthermore, the correlation between the TOP and the physical and mechanical measurements, both after artificial and natural ageing, gives further confirmation of a previous assertion about the different results after natural ageing, as compared with artificial ageing [7]. The natural decay deeply modifies the edges between grains, widens cracks and thus increases the degree of porosity. The changes in the marble, after artificial ageing, are only related to the mechanical behaviour and are not associated with the considerable increase in porosity.

Some remarks should be made on the technique used to determine TOP in comparison with the "standardized" determination of Open Porosity: the two determinations give different values as are related to different informations. The open porosity determination by means the standardized methods cannot give any information about the evolution of porosity inside the specimen, as it is referred to the whole sample. On the other side, the not "standardized" technique, when the porosity is very small, could be not representative as it could be difficult to fill all micropores with the staining agent in order to identify the void by means the image analysis.

AGA shows a poor correlation to the physical and mechanical measurements, considering both natural and the artificial ageing, apart from the water absorption. Water absorption, in fact, correlates well with AGA as well as with TOP. The correlation between AGA and WA after the two different kind of decay could be due to habit of the grain. In other words for a polygonal structure with small contact among grains the decay is linked to an increase of water absorption. Lower is the AGA and greater is the tendency to increase in porosity and then in water absorption of sample tested mainly in the artificial ageing (with a best correlation).

Finally, from the results obtained, it is possible to assert that AGA cannot be fully representative of the tendency to bowing of the marble. In fact, SG and RE, despite the AGA values, respectively 6.8 and 6 with their grains polygonal and weakly interlobate, don't show any bow tendency and decrease in mechanical properties (tables 3 and 4). Finally, further investigations could be carried out, focusing on the influence of grain size on bow tendency, and the use of Image analysis as an instrument to evaluate this feature.

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